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ANALYSIS OF THE NAVAIRDEVCEN SELF-PRIMING TOPCOAT ON GRAPHITE/EPOXY COMPOSITES

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The standard coating system for Navy aircraft consists of an epoxy-polymade primer (MIL-P-23377) under a polyurethane topcoat (MIL-C-83286). In September of 1986, the Naval Air Development Center developed a self-priming topcoat (single coating) to replace the standard Navy aircraft coating system. Equivalent or superior paint properties were achieved with potential material and labor savings. This self-priming topcoat was developed for use on aluminum-skinned aircraft. Since the amount of polymeric composite components used on naval aircraft is rapidly increasing, the current effort was undertaken to determine the compatibility of the self-priming topcoat with graphite/epoxy composite substrates.

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INTRODUCTION

The standard paint system for naval aircraft consists of a polyurethane topcoat (MIL-C-83286 or MIL-C-85285) over a epoxy-polyamide primer (MIL-P-23377 or MIL-P-85582). Occasionally, a polysulfide sealant (MIL-S-8802 or MIL-S-81733) is applied between the primer and the topcoat to increase the flexibility of this coating system in highly stressed areas such as fastener patterns. The topcoat provides the weather, abrasion, and fluid resistance, and the optical properties such as color, opacity, and gloss. The primer, which currently contains carcinogenic pigments (chromates), acts as a corrosion inhibitor and an adhesion promoter.

In September of 1986, the Naval Air Development Center developed a single coating system to replace the standard two or three coating systems described above. In effect, a self-priming topcoat (SPTC) was produced (1). Equivalent or superior pain properties were achieved with potential weight, material, and labor savings. This SPTC was developed for use on aluminum-skinned aircraft. Since the amount of graphite fiber reinforced epoxy (Gr/Ep) composite components used on Navy aircraft is rapidly increasing, the current effort was undertaken to determine the compatibility of the SPTC with Gr/Ep composite substrates.

EXPERIMENTAL

MATERIALS

The materials analyzed in this effort were the SPTC and the control coating system (MIL-P-23377/MIL-C-83286) on Gr/Ep substrates. The SPTC consisted of a two component aliphatic polyurethane resin with a non-lead, non-chromate pigment system (Table I). Specifically, the resin components are a polyester diol reacted with a hexamethylene diisocyanate. The pigment system consisted of titanium dioxide vesticulated beads (2), titanium dioxide, a proprietary organo-zinc complex, zinc molydbate, and zinc phosphate. The titanium dioxide vesticulated beads and the titanium dioxide impart the opacity and color to the SPTC while the three other pigments mainly provide corrosion inhibition but also contribute to the opacity and color. The solvent system, excluding the solvents in the resin system, consisted solely of 1,1,1-trichloroethane which is currently classified as a volatile organic compound (VOC) exempt solvent.

The control coating system consisted of an epoxy-polyamide primer (MIL-P-23377 type I) under an aliphatic polyurethane topcoat (MIL-C-83286). The primer used in this analysis contained approximately 27% strontium chromate by weight in the dry film.

The substrate material consisted of the Hercules AS/3501-6 graphite fiber/epoxy matrix composite system.

PROCEDURES

Application

The surfaces of the Gr/Ep substrates were prepared by gently wiping using non-oil extractable wipes moistened with reagent grade methyl ethyl ketone. The coating were applied using conventional air spray equipment at the desired coating thicknesses: 0.6 mils (15.4 micron) to 0.9 mils (22.86 micron) for MIL-P-23377, and 1.7 mils (43.18 micron) to 2.3 mils (58.42 micron) for both MIL-C-38286 and the SPTC. The coatings were allowed to cure for one week at ambient laboratory conditions prior to testing.

The two coating system were analyzed based on adhesion, fluid immersion, and accelerated environmental exposure properties. In the tests involving fluid and/or environmental acrylic lacquer to prevent moisture diffusion through the composite specimen to the coating/substrate interface. Also, the perimeter of the coated test panels were sealed with wax to prevent edge effects.

Adhesion

The X-cut tape and the cross-hatch tape tests (ASTM D3359, Methods A and B) were used to analyze the adhesion of the coatings to the Gr/Ep substrates (three trials per coating per test). Wet tape adhesion tests (Fed. Test Method Std. 141C, Method 6301.2) were also conducted in which the coated substrates were first subjected to 24 hours static immersion in distilled water at room temperature (75 ± 5°F) before using X-cut and cross-hatch test procedures. Accelerated wet tape adhesion tests were performed in which the temperature was 120 ± 5°F at 4 and 10 days. The dry and wet tape tests were evaluated using the classifications listed in ASTM D3359 (Tables II & III).

Fluid Immersion

The coated Gr/Ep specimens (two trials per coating per test) were exposed to a variety of fluids, at various temperatures and durations: hydraulic fluid (MIL-H-83282) at 150 ± 5°F for 1 day, lubricating oil (MIL-L-23699) at 250 ± 5°F for 1 day, jet fuel at 75 ± 5°F for 14 days, distilled water at 75 ± 5°F for 4 and 10 days. After the specimens were subjected to the appropriate test conditions, the coatings were examined for softening, blistering, uplifting or any other defects.

Accelerated Environmental Exposure

Accelerated weathering, 95% relative humidity, and 5% NaCl salt fog tests were used to determine coating/substrate resistance to these accelerated environmental conditions. In all three tests (2 trials per coating per test), to the coating were examined for blistering, uplifting, or any other coating delamination defect after being subjected to the designated test conditions. For 500 and 1000 hr xenon-arc accelerated weathering (ASTM G26), the test specimens were subjected to a constant 6000 watt light source with a water spray being introduced the last 18 minutes of every two hours. Other cabinet conditions include a black body temperature of 140 ± 5°F, a relative humidity of 50 ± 5%, and a Xe-arc intensity of 0.3 to 0.4 watt/sq. meter at 340 nm wavelength. For humidity resistance (ASTM B117), the specimens were subjected to 95% relative humidity at 120 ± 2°F for 30 days. For 2000 hr salt fog resistance (ASTM B117), the test specimens were subjected to a 5% NaCl salt fog at an orientation of 15 degrees from vertical and examined every 500 hours for coating defects and also for delamination by tape test.

RESULTS AND DISCUSSION

The objective of the current effort was to determine the compatibility of the SPTC with Gr/Ep composite substrates. The SPTC was analyzed and comparatively rated against the standard Navy coating system on Gr/Ep substrates based on three main properties: adhesion, fluid immersion resistance, and accelerated environmental resistance. Due to the inherent structural properties of Gr/Ep composites, several common physical tests were not conducted. For example, impact resistance (toughness), impact flexibility, and mandril bend flexibility tests could not be performed due to the rigidity of the Gr/Ep panels. The common corrosion resistance tests (5% NaCl salt fog, S02/NaCl salt fog, and filiform) do not yield substrate corrosion data since polymers degrade rather than corrode. However, the SPTC has been analyzed for flexibility and corrosion protection on aluminum (1). A summary of the test results for the coatings on Gr/Ep substrates is provided in Table IV.

Adhesion can be defined (as in ASTM D907) as 'the state in which two surfaces are held together by interfacial forces which consist of valence forces or interlocking forces, or both.' The force of removal can be determined by using the X-cut and cross-hatch which combine two mechanisms of removal: scraping and peeling. These two tests showed that the control coating and SPTC both possess good substrate/coating adhesion to Gr/Ep. Both coatings passed the dry and wet tape adhesion tests at the standard and accelerated conditions. The values obtained were 4A by the X-cut method and 4B by the cross-hatch method. Small areas at the fringe of the incision were removed, causing the rating to be slightly less than perfect but still passing (Figure 1).

The solutions used in the fluid immersion tests are indicative of common Navy aircraft operational fluids that may come in contact with coated Gr/Ep substrates. In an earlier study, these solutions were determined to be non-deleterious (with respect to tensile strength) to bare Gr/Ep substrates immersed for over two months at approximately 212°F (3). Also, the SPTC on aluminum was found to be resistant to these fluids at conditions identical to the current study (1). Thus the current fluid immersion tests actually indicate the permeability (or the non-permeability) of the coating to these fluids. These exposures also further test adhesion since the permeability of these fluids in these coatings may affect the coating/substrate interface. Other than some discoloration, the control coating and the SPTC were not affected by the test fluids. They showed good barrier resistance (no softening) and good adhesion (no blistering or any other coating delamination).

The xenon-arc accelerated weathering, 95% humidity, and 5% NaCl salt fog resistance tests are designed to simulate environmental conditions but at highly accelerated rates. Since Gr/Ep substrates produce no corrosion products in these environments, these tests are actually being used to determine the resistance of the coating to degradation, permeability, and its related adhesion properties. The SPTC and the control coating system showed no signs of blistering, uplifting, or any other type of coating delamination after exposure to these test conditions. Tape adhesion testing, performed on the specimens exposed to salt fog, produced no removal of the coatings.

CONCLUSIONS AND RECOMMENDATIONS

The SPTC performed as well as the control coating (MIL-P-22337/MIL-C-83286) on Gr/Ep substrates in controlled laboratory tests. After the anticipated successful completion of fleet testing (currently on Gr/Ep panels on one F-14 and three H-3's), this coating can be effectively transitioned into the fleet for use on Gr/Ep components on Navy aircraft.

It is recommended that an optimized self-priming topcoat with primary and/or barrier pigments alone be developed for use on Gr/Ep or other non-metal substrates at a future date. Since, corrosion inhibitive pigments (in general) are heavier and more expensive than barrier pigments, a reduction in coating weight and raw materials expense can be achieved. Also, a study of the disbondment characteristics of organic coatings on polymeric substrates would be beneficial to future coatings development.

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Table I: Composition Of Lusterless White Self-Priming Topcoat

Component	Formulation Wt%
Component A	
X3009-A (Coatings For Industry)	37.6
Titanium Dioxide (DuPont)	1.1
Titanium Dioxide Vesticulated Beads (Enterprise)	0.4
Zinc Molybdate (Sherwin Williams)	29.9
Zinc Phosphate (Mineral Pigments)	17.0
Sicorin RZ, an organo-zinc complex (BASF)	1.7
Anti-Terra-204 (Byk Chemie)	0.5
Sub-Total	88.2
Component B	
X3009-B (Coatings For Industry)	11.8
Total	100.0

Table II: X-Cut (Method A) Adhesion Rating

Rating	Description
5A	No peeling or removal
4A	Trace peeling or removal along incisions.
3A	Jagged removal along incisions up to 1/16 inch (1.6 mm) on either side.
2A	Jagged removal along most of incisions up to 1/8 inch (3.2 mm) on either side.
1A	Removal from most of the area of the X under the tape.
0A	Removal beyond the area of the X.

Table III: Cross-Hatch (Method B) Adhesion Rating

Rating	Description
5B	The edge of the cuts are completely smooth; none of the squares of the lattice is detached.
4B	Small flakes of the coating are detached at intersections; less than 5% of the area is affected.
3B	Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15% of the lattice.
2B	The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35% of the lattice.
1B	The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65% of the lattice.
0B	Flaking and detachment worse than Rating 1B.

Table IV: Summary Of Results On Gr/Ep

	Test Conditions	MIL-P-23377/ MIL-C-83286	Self-Priming Topcoat
ADHESION			
X-Cut:	Dry, 75°F	4A	4A
	H_2O, 75°F, 1 day	4A	4A
	H_2O, 120°F, 4 day	4A	4A
	H_2O, 120°F, 10 day	4A	4A
Cross-Hatch:	Dry, 75°F	4B	4B
	H_2O, 75°F, 1 day	4B	4B
	H_2O, 120°F, 4 day	4B	4B
	H_2O, 120°F, 10 day	4B	4B
FLUID IMMERSION			
MIL-H-83282:	150°F, 1 day	ND	ND
MIL-L-23699:	250°F, 1 day	ND	ND
JP-5:	75°F, 1 day	ND	ND
H_2O:	75°F, 1 day	ND	ND
	120°F, 4 day	ND	ND
	120°F, 10 day	ND	ND
Break Free:	75°F, 14 day	ND	ND
ACCELERATED ENVIRONMENTAL EXPOSURE			
Weatherometer:	Xe-arc, H_2O spray, 500 hr	ND	ND
	Xe-arc, H_2O spray, 1000hr	ND	ND
Humidity:	95% RH, 120°F, 30 day	ND	ND
Salt Fog:	5% NaCl, 2000 hr, tape adhesion	ND	ND

ND - No coating defects

Figure 1: X-Cut Adhesion Test On Coated Gr/Ep Subs rates

MIL-P-23377/MIL-C-83286

SELF-PRIMING TOPCOAT

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